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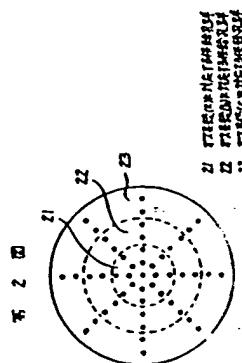
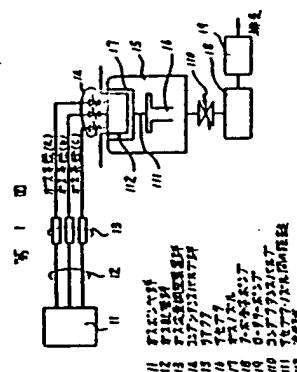
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30.03.90-JP-080739 (12.12.91) C23c-16/44 H011-21/20  
CVD appmts. giving uniform thickness films - has concentric gas feed  
nozzles divided into radial gps. and facing susceptor  
C92-016059

Concentrically distributed gas feeding nozzles are faced to the susceptor in the reactor. The nozzles are divided into radial groups, where gas flow rate and conductance are controlled per each group individually.

ADVANTAGE - Uniform film thickness is obtd.. (4pp  
Dwg No. 1.2/5)

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-1- (WPAT)

ACCESSION NUMBER  
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TITLE

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CVD appts. giving uniform thickness films - has concentric gas feed nozzles divided into radial gps. and facing susceptor

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(J03281780)

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ADVANTAGE - Uniform film thickness is obtd..

(4pp Dwg. No. 1,2/5)

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IMAGE FILENAME

-2- (JAPIO)

ACCESSION NUMBER

TITLE

PATENT APPLICANT

INVENTORS

PATENT NUMBER

APPLICATION DETAILS

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ABSTRACT

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CVD DEVICE

(2000510) HITACHI LTD

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C23C-016/44, C23C-016/52, H01L-021/205, H01L-021/285

12.6 (METALS--Surface Treatment), 42.2

(ELECTRONICS--Solid State Components)

PURPOSE: To obtain a uniform gas flow in accordance with the pressure region of the gas in a wide range and the temp. range of a susceptor by providing a function which regulates the flow rate of gas and conductance at every group of a plurality of nozzles opposite to the susceptor.

CONSTITUTION: The gas supply system of a CVD device is constituted of a plurality of gas pipeline groups 12, flow rate controlling device groups 13 and conductance valve groups 14. A gas nozzle 17 opposite to a susceptor 16 is equipped in a reactor 15. The gas supply face of the gas nozzle 17 has a plurality of aperture groups 21-23 for supplying gas, which are distributed in a concentrical circular shape. These aperture groups are allotted to a plurality of kinds of gas system (a)-(c). Respective gas systems (a)-(c)

## ⑫ 公開特許公報 (A) 平3-281780

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 7739-4M  
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## ⑤発明の名称 CVD装置

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 ②出 願 平2(1990)3月30日

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PTO 98-2891

S.T.I.C. Translations Branch

## 明細書

## 1. 発明の名称

CVD装置

## 2. 特許請求の範囲

1. CVD装置において、反応管内でサセプタと対向しているガス供給ノズル部を有し、該ノズルは同心円状に分布しており、且つ該半径方向に複数に分割したノズル群毎にガス流量及びコンダクタンスを制御できることを特徴とするCVD装置。

2. 複数の反応ガスを混合した後、上記同心円状に分布したノズルによりガスをサセプタ側に供給することを特徴とする請求項1記載のCVD装置。

3. 上記半径方向に複数に分割したノズル群毎に膜厚センサを具備しており、上記各ノズル群毎にガス流量及びコンダクタンスについてフィードバックをかけることが可能であることを特徴とする請求項1記載のCVD装置。

4. 上記半径方向に複数に分割したノズル群毎に

ガス圧センサを具備しており、上記各ノズル群毎にガス流量及びコンダクタンスについてフィードバックをかけることが可能であることを特徴とする請求項1記載のCVD装置。

## 3. 発明の詳細な説明

## (産業上の利用分野)

本発明はCVD装置に係り、特に広い圧力範囲にわたって膜厚の面内均一性が良好な膜を成膜できる減圧CVD装置に関する。

## (従来の技術)

供給律速系のCVD法においては、面内の膜厚均一性は試料表面における各反応ガス分圧の分布の影響を受けることが知られている。その対策として、例えばJournal of Crystal Growth 77 (1986) 151-156に記載されているように、試料表面すなわちサセプタに対して均一なガスフローが得られるように反応管形状の最適化を図り、試料面内における反応ガス分圧の均一化を達成している。

## (発明が解決しようとする課題)

上述した反応管形状、サセプタ形状及び両者間の距離による幾何学的なガスフローの均一化は、特定の条件下においてのみ有効で、全ガス圧やサセプタ温度の影響を受けるので汎用的とは言い難い。本発明の目的は広範囲なガス圧領域、サセプタ温度領域に対応して均一なガスフローを得ることが可能なCVD装置を提供することにある。

【課題を解決するための手段】

CVD装置において、反応管内にサセプタと対向し且つ同心円状に分布したガスノズルを設け、該半径方向に分割した複数のノズル群毎にガス流量及びコンダクタンスを調整できる機能を有するCVD装置を採用することにより上記課題を解決した。

【作用】

円筒型反応管においてガスの流れに垂直に置かれたサセプタ上におけるガス分圧の分布は同心円状の分布になつていて、そこでサセプタと対向し同心円状に分布したガスノズルを用い、ガス流量及びコンダクタンスを複数の同心円状ノズル群毎

に調整して、前記ガス分圧の分布を補正し、均一化を図ることが可能なCVD装置を用いることにより、広範囲のガス圧力下及び広範囲の温度領域において試料表面におけるガス圧を均一にすることができる。その結果、CVD膜厚の面内分布が良好になる。

【実施例】

実施例1

本発明の実施例1を第1図に示したCVD装置の概略図及び第2図に示したガスノズルの供給面側の形状図より説明する。CVD装置の基本構成はガス供給系、リアクタ、ガス排気系よりなつていて、ガス供給系はガスポンベ11と複数の（ここでは3系統）ガス配管群12と該各配管に対応する流量制御装置群13及びコンダクタンスバルブ群14より構成されている。

リアクタ15内には加熱が可能なサセプタ16と対向したガスノズル17が具備されている。ガスノズルのガス供給面は第2図に示したように円状であり同心円状に分布した複数の細孔よりなる

ガス供給用開孔部を有している。同心円状に分布したガス供給用開孔群は3種類のガス系統(a)、(b)、(c)に割り当ててガス系統(a)に対応する供給孔群21、ガス系統(b)に対応する供給孔群22、ガス系統(c)に対応する供給孔群23に分類できる。該供給孔群を流れるガスは各々独立に流量制御、コンダクタンス制御を行つていて、排気系はターボ分子ポンプ18とロータリポンプ19より構成され、コンダクタンスバルブ110を用いてリアクタ15内の圧力を調整している。

ガス系統(a)にはWF<sub>6</sub>を2SCCM、ガス系統(b)にはSiH<sub>4</sub>を1SCCM及びガス系統(c)にはH<sub>2</sub>を10SCCM各々のガス流量制御装置13（マスフローコントローラ：日本タイランFC260）及びコンダクタンスバルブ14を通して上記ガスノズル17からコールドウオール型リアクタに流した。サセプタ16は抵抗加熱により280～320℃の間で制御した。またサセプタ16とガスノズル17間の距離111

は5～15mmとした。ガス排気系はターボ分子ポンプ（アルカテル5150、排気速度140ℓ/s）18と補助ポンプとしてロータリポンプ19を用いた。このときの全ガス圧力範囲は0.05～0.1torrである。

上記CVD装置に試料としてSi基板（5インチ）を投入した場合、SiH<sub>4</sub>によるWF<sub>6</sub>の還元反応が起りW膜が堆積する。この時のW膜厚は、試料表面におけるSiH<sub>4</sub>ガス圧にほぼ比例することが知られている。SiH<sub>4</sub>ガスを1分間流した場合、5インチSiウエフ上で平均膜厚3320Å、面内分布±3%以下であった。

また該ノズルは冷却水112を循環させることにより強制的な冷却を行つていて、これによりサセプタ上の試料のみが加熱されるので、W堆積反応は試料表面に限定される。その結果第3図(a)に示したようなSiO<sub>2</sub>膜32から部分的に露出したSi31に対して選択的にWの堆積反応が起ころ。この技術を用いると第3図(b)に示したようなW膜33による選択的な孔埋め込みが可能

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#### 実施例2

実施例1では反応ガスの混合をガスノズルから流出させた後リアクタ内で行つてはいる。本実施例では第4図に示すように各々のガス流量制御装置群42を通した後、ガス混合用配管43を通して反応ガスの混合を行つた。その際、ガスの逆流防止弁を適所に設置したことは言うまでもない。然る後、コンダクタンスバルブ群44及び第2図と同様のガスノズル45を通して反応ガスをリアクタ47に導入した。その他のCVD条件は実施例1と同じで同様な結果を得た。本実施例を用いると各反応ガス流量に対応させて各反応ガス用コンダクタンスバルブを調整する煩雑さが無くなる。この場合、全反応ガス流量のみに注目して各ガスノズル開孔部分に対応してコンダクタンスバルブを調整すれば良い。またサセプタ46とガスノズル45間の距離411が成膜特性に与える影響が小さい。

本実施例では全反応ガス流量と全反応ガスの圧

試料表面における反応ガスの分圧を均一にすることができるので膜厚の均一なCVD膜の形成が可能になる。本発明は選択CVD等、特に高真空下においてCVDを行う場合極めて有効である。

#### 4. 図面の簡単な説明

第1図は本発明の実施例1のCVD装置構成図、第2図は本発明のガスノズルのガス供給面側の一形状図、第3図(a)及び第3図(b)は実施例1の選択CVD説明図、第4図は実施例2におけるCVD装置構成図、第5図は実施例3のCVD装置構成図である。

11, 42, 51…ガス配管群、13, 42, 52…ガス流量調整装置群、14, 44, 54…コンダクタンスバルブ群、16, 46…サセプタ、15, 47…リアクタ、43…ガス混合用配管、17, 45, 55…ガスノズル、21…ガス系統(a)に対応する供給孔群、22…ガス系統(b)に対応する供給孔群、23…ガス系統(c)に対応する供給孔群、111, 411…サセプタとノズル間の距離、56…圧力センサ、57…膜厚センサ、510…制御用コンピュータ、

特開平3-281780(3)

力に応じてガス供給系のコンダクタンスバルブ並びにサセプタとノズル間の距離を調整する必要がある。これらの調整をコンピュータ制御により自動化を図ることが可能であることは言うまでもない。

#### 実施例3

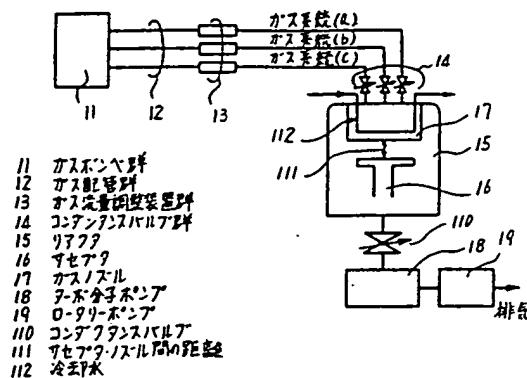
上述した実施例では全反応ガス流量と全反応ガスの圧力に応じてガス供給系のコンダクタンスバルブ並びにサセプタとノズル間の距離等のパラメータを設定し開ループ制御で膜形成を行つてはいる。本実施例では第5図に示すようにガスノズル55のガス供給面半径方向に複数の圧力センサ56及び複数の膜厚センサ57を具備させて非破壊で試料表面近傍のガス圧並びにCVD膜厚をモニタしてガス流量調整装置52並びにコンダクタンスバルブ54にフィードバックをかけてW膜を形成した。本実施例を用いて5インチウェーフ上でのW膜厚分布を±0.5~1.0%以下に低減できた。

#### 【発明の効果】

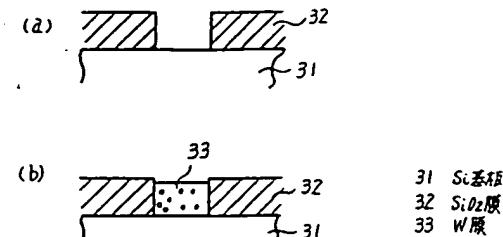
本発明を用いると、広範囲なガス圧力に対して

代理人 井理士 小川勝男

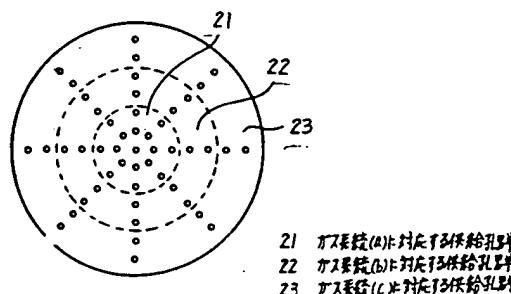
第1図



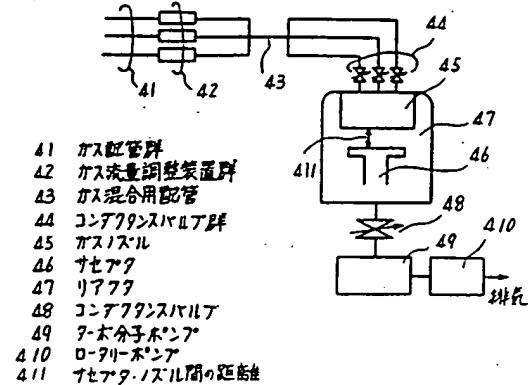
第3図



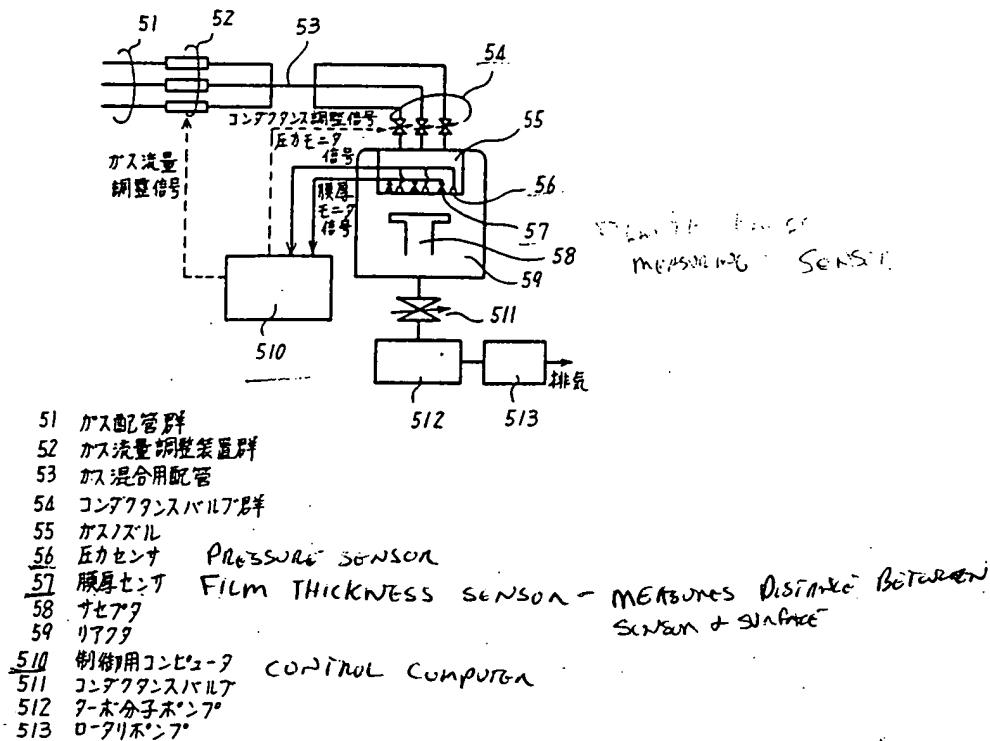
第2図



第4図



第5図



PTO 98-2891

CY=JP DATE=19911212 KIND=A  
PN=03281780

A CVD DEVICE  
[CVD sōchi]

Katsuhiko Mitani et al.

UNITED STATES PATENT AND TRADEMARK OFFICE  
Washington, D.C. May 1998

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HIROSHI  
APPLICANT (71) : HITACHI, LTD.  
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FOREIGN TITLE [54A] : CVD SŌCHI

## Specification

### 1. Title of the Invention

A CVD device

### 2. Claims

1. A CVD device, said CVD device characterized as having a gas feeding nozzle part that faces a susceptor inside a reaction tube, and by the fact that said nozzles are distributed in a concentric state, and the gas feeding amount and conductance can be controlled for each one of several nozzle groups that are distributed in the radial direction.

2. A CVD device according to Claim 1, characterized by the fact that, after multiple reaction gases have been blended, the gas is supplied to the susceptor side from the aforesaid concentrically distributed nozzles.

3. A CVD device according to Claim 1, characterized by the fact that a film thickness sensor is provided for each of the aforesaid multiple nozzle groups distributed in the radial direction, allowing feedback to be provided with regard to the gas flow amount and conductance for each of the aforesaid nozzle groups.

4. A CVD device according to Claim 1, characterized by the fact that a gas pressure sensor is provided for each one of the aforesaid multiple nozzle groups distributed in the radial direction, allowing feedback to be performed with regard to the gas flow amount and conductance for each of the aforesaid nozzle groups.

### 3. Detailed Explanation of the Invention

#### (Industrial Field of Application)

The present invention relates to a CVD device, in particular an *in vacuo* CVD device that can form a good film having in-plane uniformity within a wide range of pressures.

#### (Prior Art)

In feed rate determining-type CVD methods, it is known that the in-plane uniformity of film thickness is subject to the effects of the distribution of the partial pressures of each reaction gas on the sample surface. As countermeasures against this, for example, as specified in the Journal of Crystal Growth 77 (1986), 151-156, the optimization of the reaction tube formed is planned so as to obtain a uniform gas flow with regard to the sample surface, i.e., the susceptor, and the equalization of reaction gas partial pressure within the sample flame is achieved.

#### (Problems that the Invention is to Solve)

The geometric equalization of the gas flow due to the aforesaid reaction tube formed, susceptor formed, and distance between the two is effective only under specified conditions, and is susceptible to the effects of total gas pressure and susceptor temperature, and thus cannot be said to be universal. The present invention has the purpose of offering a CVD device that is able to obtain a uniform gas flow with regard to a wide range of gas pressure regions and susceptor temperature regions.

#### (Means of Solving the Problems)

The aforesaid problems are solved by means of a CVD device, said

CVD device characterized as having a gas feeding nozzle part that faces a susceptor inside a reaction tube, and by the fact that said nozzles are distributed in a concentric state, and the gas feeding amount and conductance can be controlled for each one of several nozzle groups that are distributed in the radial direction.

(Operation)

The distribution of gas partial pressure on a susceptor that is placed perpendicular to the flow of gas in a cylindrical reaction tube is a concentric distribution. Thus, by using a CVD device that uses gas nozzles that are distributed concentrically with regard to the susceptor, and that by adjusting the gas flow amount and conductance with regard to each of multiple concentric nozzle groups is able to correct and equalize the distribution of the aforesaid gas partial pressure, it is possible to equalize the gas pressure on the sample face in a wide range of gas pressures and a wide range of temperature regions. As a result, the in-plane distribution of the CVD film thickness is improved.

(Working Examples)

Working Example 1

Working Example 1 of the present invention is explained beginning with the schematic drawing of a CVD device shown in Figure 1 and the form drawing of the gas nozzle feeding face side shown in Figure 2. The CVD device is basically composed of a gas feeding system, a reactor, and a gas exhaust system. The gas feeding system consists of a gas cylinder (11), multiple gas piping groups (12) (in this case 3 systems), and flow control device groups (13) and conductance valve groups (14).

corresponding to said pipings.

A heatable susceptor (16) and corresponding gas nozzle (17) are placed in the reactor (15). The gas feeding face of the gas nozzles has a gas feeding hole part composed of multiple fine holes arranged in a circular and concentric form as shown in Figure 2. The gas feeding hole groups that are arranged concentrically are allotted into 3 types of gas systems (a), (b), and (c) and are classified into feeding hole group (21) corresponding to gas system (a), feeding hole group (22) corresponding to gas system (b), and feeding hole group (23) corresponding to gas system (c). Flow amount control and conductance control are performed independently for the gas that flows through each of said feeding hole groups. The exhaust system is composed of a turbomolecular pump (18) and rotary pump (19) and adjusts the pressure in the reactor (15) using a conductance valve (110).

In the gas system (a) 2 SCCM of  $WF_6$ , in the gas system (b) 1 SCCM of  $SiH_4$ , and in the gas system (c) 10 SCCM of  $H_2$  were passed through the gas flow amount control device (13) (mass controller: Nippon Tairan FC260) and conductance valve (14) from the aforesaid gas nozzle (17) into a cold wall-type reactor. The susceptor (16) was controlled so as to be within a range of 280 to 320°C by resistance heating. The distance (111) between the susceptor (16) and gas nozzle (17) was made 5 to 15 mm. The gas exhaust system used a turbomolecular pump (Arukateru [as transliterated] 5150, gas exhaust speed 140 l/s) (18) and a rotary pump (19) as an auxiliary pump. The total gas pressure range at this time was 0.05 to 0.1 Torr.

When a Si substrate (5 inches in diameter) was inserted into the

aforesaid CVD device as a test material, a reduction reaction of  $WF_6$  caused by  $SiH_4$  occurred, and a W film was deposited. It is known that the thickness of the W film at this time is approximately proportional to the  $SiH_4$  gas pressure on the sample surface. When  $SiH_4$  gas was allowed to flow for 1 min, an average film thickness of 3320 Å with an in-plane distribution of  $\pm 3\%$  or less was formed on a 5-inch diameter Si wafer.

In addition, forced cooling was performed on said nozzle by the circulation of cooling water (112). By this means, only the example on the susceptor was heated, so the W deposition reaction was limited to the sample surface. As a result, a deposition reaction of W occurred selectively with regard to Si (31), which was partially exposed from the  $SiO_2$  film (32) as shown in Figure 3(a). Using this technique, selective hole fill-in by means of a W film (33) as shown in Figure 3(b) becomes possible.

#### Working Example 2

In Working Example 1, the mixture of reaction gases was performed inside the reactor after the gases were discharged from the gas nozzle. In this working example, after passing through the respective gas flow control device groups (42), as shown in Figure 4, the gases were passed through the gas blending tubes (43) and blending of the reaction gases was performed. In this process, needless to say, gas flow check valves were installed in suitable locations. Next, the reaction gas was passed through the conductance valve group (44) and a gas nozzle (45) similar to that shown in Figure 2 and introduced into the reactor (47). The other CVD conditions were the same as those in Working Example 1, and

similar results were obtained. Using this working example, the complexity of adjusting the conductance valve for each reaction gas in response to each reaction gas flow amount is eliminated. In this case, the conductance can be adjusted in correspondence with each gas nozzle opening part considering only the total reaction gas flow amount. Moreover, the effects of the distance (411) between the susceptor (46) and gas nozzle (45) on the film formation characteristics are small.

In this working example, it is necessary to adjust the conductance valve of the gas feeding system and the distance between the susceptor and the nozzle in accordance with the total reaction gas flow amount and total reaction gas pressure. Needless to say, this adjustment can be automated by means of computer control.

#### Working Example 3

In the working examples described above, film formation is performed by open-loop control whereby parameters such as the conductance valve in the gas feeding system and the distance between the susceptor and nozzle are set in accordance with the total reaction gas flow amount and total reaction gas pressure. In the present working example, as shown in Figure 5, multiple pressure sensors (56) and multiple film thickness sensors (57) are installed in the radial direction of the gas feeding surface of the gas nozzle (55), and the gas pressure in the vicinity of the sample surface and the CVD film thickness are non-destructively monitored, providing feedback to the gas flow amount control device (52) and conductance valve (54) to form a W film. Using this working example, it was possible to reduce the W film thickness distribution on a 5-inch diameter wafer to  $\pm 0.5$  to 1.0% or

less.

(Effects of the Invention)

Using the present invention, it is possible to equalize the distribution of reaction gas on the sample surface with regard to a wide range of gas pressures, so that the formation of a CVD film having uniform film thickness becomes possible. The present invention is extremely effective when selective CVD, etc. and particularly CVD is performed in a high-level vacuum.

4. Brief Explanation of the Figures

Figure 1 is a structural diagram of a CVD device in Working Example 1 of the present invention, Figure 2 is a formal diagram of the gas feeding surface side of the gas nozzle in the present invention, Figure 3(a) and Figure 3(b) are diagrams explaining selective CVD in Working Example 1, Figure 4 is a structural diagram of the CVD device in Working Example 2, and Figure 5 is a structural diagram of the CVD device in Working Example 3.

11, 42, 51: gas piping group, 13, 42, 52: gas flow amount adjustment device group, 14, 44, 54: conductance valve group, 16, 46: susceptor, 15, 47: reactor, 43: gas mixing tube, 17, 45, 55: gas nozzle, 21: feeding hole group corresponding to gas system (a), 22: feeding hole group corresponding to gas system (b), 23: feeding hole group corresponding to gas system (c), 111, 411: distance between susceptor and nozzle, 56: pressure sensor, 57: film thickness sensor, 510: control computer

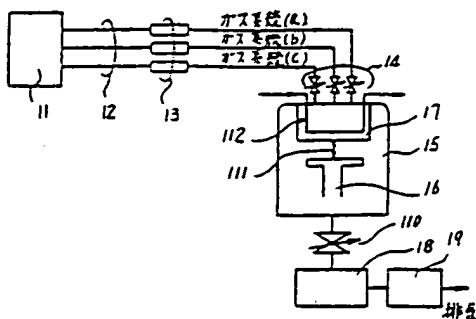


Figure 1  
gas system (a)

gas system (b)

gas system (c)

[bottom right:] exhaust

11: gas canister group

12: gas piping group

13: gas flow amount adjustment device group

14: conductance valve group

15: reactor

16: susceptor

17: gas nozzle

18: turbomolecular pump

19: rotary pump

110: conductance valve

111: distance between susceptor and nozzle

112: cooling water

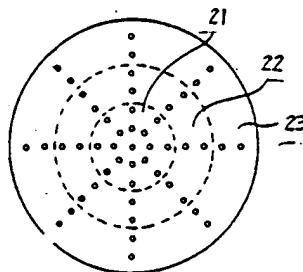


Figure 2

21: feeding hole group corresponding to gas system (a)

22: feeding hole group corresponding to gas system (b)

23: feeding hole group corresponding to gas system (c)

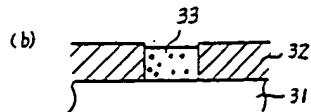
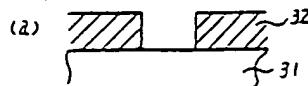


Figure 3

31: Si substrate

32:  $\text{SiO}_2$  film

33: W film

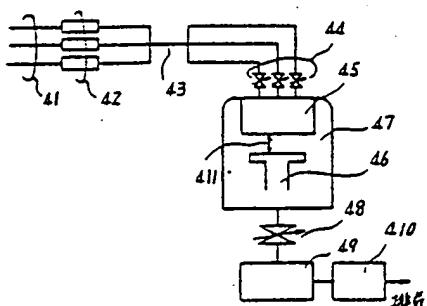


Figure 4

41: gas piping group

42: gas flow amount adjustment device group

43: gas mixing tube

44: conductance valve group

45: gas nozzle

46: susceptor

47: reactor

48: conductance valve

49: turbomolecular pump

410: rotary pump

411: distance between susceptor and nozzle

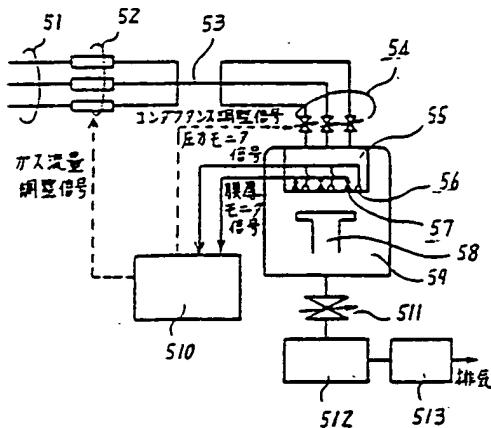


Figure. 5

[under 51:] gas flow amount adjustment signal  
 [under 53:] conductance adjustment signal  
 pressure monitor signal  
 film thickness monitor signal

[right of 513:] exhaust

- 51: gas piping group
- 52: gas flow amount adjustment device group
- 53: gas mixing tube
- 54: conductance valve group
- 55: gas nozzle
- 56: pressure sensor
- 57: film thickness sensor
- 58: susceptor
- 59: reactor
- 510: control computer
- 511: conductance valve
- 512: turbomolecular pump
- 513: rotary pump